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IS 13842 (1993): m-Ureidoaniline, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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भारतीय मानक

एम-यूरीडोनीलीन, तकनीकी – विशिष्टि

Indian Standard

m-UREIDOANILINE, TECHNICAL — SPECIFICATION

UDC 667·281·26

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1993

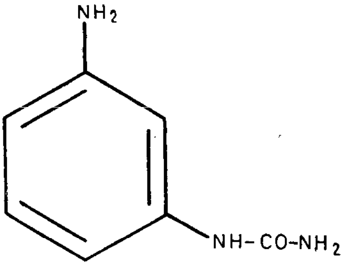
Price Group 2

Dye Intermediates Sectional Committee, PCD 11

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

m Ureidoaniline ($C_7H_9N_3O$) is used as an intermediate for reactive dyes. It is represented by the following structural formula:



m-UREIDOANILINE

(MOLECULAR MASS 151)

CAS REGISTRY NO. (25711-72-2)

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off values should be the same as that of the specified values in this standard.

Indian Standard

m-UREIDOANILINE, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for *m*-ureidoaniline.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1992	Water for general laboratory use (*third revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) (*third revision*)
5299 : 1969	Methods of sampling and tests for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be moist, yellowish-brown to grey or pale purple in colour.

3.2 The material shall also comply with the requirements given in Table 1

Table 1 Requirements for *m*-Ureidoaniline, Technical

(*Clause* 3.2)

Sl No.	Characteristic	Requirements	Methods of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Matter insoluble in water (on 100 percent basis), percent by mass, *Max*	10·0	A
ii)	Assay, percent by mass, *Min*	45·0	B
iii)	*m*-Phenylenediamine (on 100 percent basis), percent by mass, *Max*	0·50	C

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552 : 1989) or HDPE bags lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

4.2 Marking

Each container shall bear legibly and indelibly the following information:

a) Name of the material;

b) Indication of the source of manufacture;

c) Tare, net and gross mass;

d) Batch number; and

e) Month and year of manufacture.

4.2.1 The containers may also be marked with the Standard Mark.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in 3 of IS 5299 : 1969.

5.2 Number of Tests

5.2.1 Test for assay and determination of *m*-phenylenediamine shall be conducted on each of the individual samples.

5.2.2 Tests for the determination of remaining characteristics, namely, description and matter insoluble in water shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirement of assay and phenylenediamine content if each of the individual test result satisfies the relevant requirement given in Table 1.

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics (*see* **5.2.2**) tested on the composite sample, the test results for each of the characteristic shall satisfy the relevant requirements given in **3.1** and Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods referred in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis'.

ANNEX A

[*Table* 1, *Sl No.* (i) and *Clauses* A-1 *and* A-2]

DETERMINATION OF MATTER INSOLUBLE IN WATER

A-1 PROCEDURE

Weigh accurately about 30 g of the sample and transfer it to a 1-litre beaker. Add 500 ml of water stir mechanically and warm up to 35°C. Stir for 15 minutes and filter through counterpoised papers and wash the residue with 300 ml cold water. Dry the residue at 90 to 100 °C till constant mass. Preserve the filtrate and washings for assay (*see* Annex B).

A-2 CALCULATION

$$\text{Matter insoluble in water, (100 percent basis), percent by mass} = \frac{M_s \times 10\,000}{M_z \times A}$$

where

M_s = mass in g of residue;

M_z = mass in g of sample taken; and

A = assay, percent by mass (*see* Annex B)

ANNEX B

[*Table* 1, *Sl No.* (ii)]

ASSAY

B-1 REAGENTS

B-1.1 Hydrochloric Acid — Concentrated

B-1.2 Potassium Bromide

B-1.3 Sodium Nitrite Solution — 0·1 N.

B-1.4 Potassium Starch Iodide Paper

B-2 PROCEDURE

Transfer the filtrate and washings obtained in determining matter insoluble in water (*see* **A-1**) into one-litre volumetric flask and dilute to mark with water at 20°C and shake well. Pipette out 50 ml aliquot in a one-litre beaker. Add 400 ml ice cold water, 25 ml concentrated hydrochloric acid and 5·0 g of potassium bromide. Titrate, while stirring mechanically, with standard sodium nitrite, solution at 15-20°C using potassium starch iodide paper to a 3 minute end point.

B-3 CALCULATION

$$\text{Assay, percent by mass} = \frac{V \times N \times 302}{M}$$

where

V = volume in ml of sodium nitrite solution used,

N = normality of standard sodium nitrite solution (**B-1.3**), and

M = mass in g of sample taken for the test.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF *m*-PHENYLENEDIAMINE

C-1 OUTLINE OF THE METHOD

m-phenylenediamine in *m*-ureidoaniline is determined by thin layer chromatography.

C-2 APPARATUS

C-2.1 Chromatographic Chamber — 220 mm × 100 mm × 220 mm

C-2.2 Micropipette — of 10-μl and 5-μl capacity

C-2.3 Thin Layer Chromatographic Glass Plates — 200 mm × 200 mm

C-2.4 Thin Plate Applicator — Adjustable

C-2.5 Chromatographic Spray Bottle

C-2.6 Oven

C-2.7 Weighing Bottle

C-3 REAGENTS

C-3.1 Silica Gel G — neutral

C-3.2 *m*-phenylenediamine Solution — 0·02 percent in methanol.

C-3.3 Eluent — chloroform : ethanol (80 : 20).

C-3.4 Spray Solution — 0·5 percent in 2N HCl and methanol in equal proportion.

C-4 PROCEDURE

C-4.1 Preparation of the Chromatographic Plates

Mix 50 g of neutral silica gel G with 85 to 90 ml of water for three minutes. Then, by means of the applicator, coat the plate with silica gel layer of thickness not exceeding 0·5 mm and place it in the oven maintained at 110°C for 90 minutes. By means of a needle, mark a thin line widthwise on all plates at about 3 cm from the upper edge. Also remove a band of about 5 mm width silica gel from either of the lateral sides of each plate.

C-4.2 Preparation of Sample Solution

Homogenize the sample for a few minutes and weigh by means of a weighing bottle 2·0 g 100 percent basis of the material and transfer to a 100-ml volumetric flask. Dissolve in methanol and make up the solution to the mark by adding ammonia.

C-4.3 Spot 10-μl of the sample solution (**C-4.2**) and 5-μl of the reference solution (**C-3.2**) on the thin layer plate. Place the developer in the chamber. Close the chamber with its lid and allow to achieve equilibrium. Now place the plate carefully in the chamber and allow the eluent to run in ascending manner for about 100 minutes. Remove the plate from the chamber and dry off the solvent completely. Spray the dried plate with spray solution (**C-3.4**) and heat it to 100°C.

C-4.3.1 By visual comparison, the spot corresponding to *m*-phenylenediamine in the sample shall have intensity equal to or less than that of reference sample.

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices :		Telephone
Central :	Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	331 01 31 331 13 75
Eastern :	1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola CALCUTTA 700054	37 84 99, 37 85 61 37 86 26, 37 86 62
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